## CLAIMS

What is claimed is:

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- A separation capillary column, or channel in a microfabricated device, said column or channel comprising:
- a monolithic separation medium comprising a macroporous, rigid, continuous polymeric structure, wherein said polymeric structure is attached covalently to the wall of said column or channel, wherein said column or channel has an i.d. of 25 µm or less, wherein the efficiency of operation of said column or channel is greater than 100,000 theoretical plates per meter and wherein the reproducibility of retention time on comparable said columns or channels during use varies less than 10%.
- 15 2. The capillary column or channel of claim 1, wherein the reproducibility of retention time on comparable said columns or channels during use varies less than 5%.
- 3. The capillary column or channel of claim 1, wherein said column or channel has an i.d. of 10 µm or less.
  - 4. The capillary column or channel of claim 1, wherein the efficiency of operation of said column or channel is greater than 200,000 theoretical plates per meter
  - 5. A method of preparing a separation capillary column or channel in a microfabricated device, said column or channel comprising a polymeric monolithic separation medium, said method comprising the steps of:
- providing an unfilled capillary column, or channel in a microfabricated device, said column or channel being open at both

ends thereof, the inner surface of said column or channel being suitable for covalent attachment of a polymeric monolithic separation medium;

adding to said column or channel a degassed polymerization mixture comprising monomer, crosslinking agent and inert porogens;

polymerizing said mixture in the presence of an initiator in said column or channel, during which polymerization said mixture is continuously maintained under positive pressure applied from the open ends of said column or channel; and

- following said polymerization step, washing the rigid polymeric monolithic separation medium so formed inside said column or channel to remove said porogens and any remaining polymerization mixture.
- 15 6. The method of claim 5, wherein said polymerization mixture comprises styrene monomers.
  - 7. The method of claim 6, wherein said crosslinking agent is divinylbenzene.
  - 8. The method of claim 5, wherein said polymerization mixture comprises methacrylate monomers.
- 9. The method of claim 5, wherein said column or channel has an i.d. of 40-50  $\mu m_{\odot}$ 
  - 10. The method of claim 5, wherein said column or channel has an i.d. of 20-25  $\mu m_{\cdot}$
- 30 11. The method of claim 5, wherein said column or channel has an i.d. of 10-15  $\mu m$  or less.

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- 12. The method of claim 9, wherein said positive pressure applied during polymerization is 10-20 psi.
- 5 13. The method of claim 10, wherein said positive pressure applied during polymerization is 30-60 psi.
  - 14. The method of claim 11, wherein said positive pressure applied during polymerization is 75-150 psi.

15. A process of carrying out a chemical analysis method comprising the steps of:

providing the separation capillary column, or channel in a microfabricated device, according to claim 1;

15 coupling said column or channel to a concentration sensitive detector; and

carrying out said chemical analysis method.

16. A process of carrying out a chemical analysis method 20 comprising the steps of:

providing a separation capillary column, or channel in a microfabricated device, prepared according to the method of claim 5;

coupling said column or channel prepared by said method to a concentration sensitive detector; and

carrying out said chemical analysis method.

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